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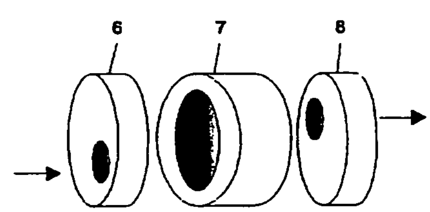
(54) **Process and apparatus for mixing or dispersing liquids**

(57) A method for mixing or dispersing liquids in which the liquids to be mixed or to be dispersed are pumped at temperatures of about 20°C to about 250°C, preferably of about 20°C to about 200°C, and pressures of about 50 bar to about 2500 bar, preferably of about 100 bar to about 800 bar, through a mixing device which consists of one or more inlet nozzles (6), one or more turbulence chambers (7) and one or more outlet nozzles (8), with the inlet nozzle(s), turbulence chamber(s) and outlet nozzle(s) being pressed in sequence in a cylindrical support and the axes of the bores of the inlet nozzle and the outlet nozzle being arranged axially to one another, as well as mixing devices for performing this method.

Fig. 3:



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Description

[0001] The present invention is concerned with a method for mixing or dispersing liquids, especially a method for the production of a finely divided liquid dispersion, as well as mixing devices for carrying out the process.

[0002] European Patent Publication EP 0776 997 A1 describes a method for the production of a finely divided dispersion of solids in which a pre-dispersion is pumped through one or more slotted nozzles. The particle size of the dispersed phase lies in the region of 0.01 μm to 20 μm. The diameter of the nozzle bore is 0.05 mm to 1 mm. The ratio of bore length to bore diameter is 1:1 to 1:10. A preferred combination comprises a device which has two nozzle bodies with the nozzles lying opposite their outlet. Also described are devices in which the crude dispersion or pre-dispersion is pumped through two or more nozzles having an equal or decreasing bore diameter. The slotted nozzle suitably consists of a ceramic material, for example, of zirconium oxide, or of metal coated with ceramic.

[0003] In International Patent Publication WO 97/17946 there is described a method for the production of a liposome dispersion in which an aqueous pre-dispersion of one or more amphiphilic substances is pumped at 600 bar to 900 bar through at least one homogenizing nozzle having a diameter of 0.1 mm to 0.5 mm. The homogenizing nozzle has an inlet channel and an outlet channel and consists of a hard ceramic plate, in which the bore is situated, pressed in a steel body. The inlet channel and the outlet channel are also incorporated in the steel body. When several nozzles are used, these are arranged opposite and have a parallel inflow. The pre-dispersion is pumped in the circuit through the homogenizing nozzle until the average particle size of the liposome dispersion lies between about 35 nm and about 80 nm.

[0004] The problem which forms the basis of the present invention is to provide a method for mixing or dispersing liquids which permits an improved intermixing with lower energy expenditure compared with the state of the art.

[0005] The problem is solved in accordance with the invention by pumping the liquids to be mixed or to be dispersed at temperatures of about 20°C to about 250°C, preferably of about 20°C to about 200°C, and pressures of about 50 bar to about 2500 bar, preferably of about 100 bar to about 800 bar, through a mixing device which consists of one or more inlet nozzles, one or more turbulence chambers and one or more outlet nozzles, with the inlet nozzle(s), turbulence chamber(s) and outlet nozzle(s) being pressed in sequence in a cylindrical support and the axes of the bores of the inlet nozzle and the outlet nozzle being arranged axially to one another.

[0006] The process is especially suitable for the production of finely divided dispersions having average particle sizes of about 10 nm to about 1000 nm, preferably of about 50 nm to about 400 nm.

[0007] For the production of liquid dispersions, a pre-emulsion is pumped at temperatures of about 20°C to about 250°C, preferably of about 20°C to about 200°C, and pressures of about 50 bar to about 2500 bar, preferably of about 100 bar to about 800 bar, through the aforementioned mixing device (dispersing unit).

[0008] The residence time of the liquids to be mixed or to be dispersed in the mixing device is about 10^{-6} sec to about 10^{-1} sec.

[0009] The term "pre-emulsion" denotes one of the following systems:

a) oil-in-water emulsion (O/W emulsion),

b) water-in-oil emulsion (W/O emulsion),

c) oil-in-water emulsion in which a lipophilic active substance is dissolved in the oil,

d) water-immiscible solvent-in-water emulsion in which a lipophilic active substance is dissolved in this solvent.

[0010] An oil-in-water emulsion in which the viscosity of the dispersed phase is about 0.01 mPas to about 10,000 mPas, preferably about 0.1 mPas to about 2000 mPas, is preferred.

[0011] The term "lipophilic active substance" embraces the vitamins A, D, E and K, carotenoids or food additives such as PUFAs (polyunsaturated fatty acids) and tocotrienols.

[0012] Advantageously, for the production of the pre-emulsion the liquid to be dispersed is stirred into an aqueous emulsifier solution, optionally while warming.

[0013] Processes for the production of finely divided liquid dispersions relate not only to processes used in the food manufacturing field and in which corresponding food emulsifiers are used, but also to general industrial dispersion processes in which corresponding industrial emulsifiers are used. Processes which are used in the food manufacturing field are preferred.

[0014] Suitable emulsifiers/stabilizers for dispersions which can be added to foods are, for example, ascorbyl palmitate, lecithins, polysorbates, sugar esters, fatty acid esters, citric acid esters, sorbitol stearates; as well as colloids, for example gelatines and fish gelatines; carbohydrates, for example starches and starch derivatives such as dextrin, pectin or gum arabic; milk proteins and plant proteins. Mixtures of the aforementioned substances can also be used. Ascor-

byl palmitate, fish gelatines or starch derivatives are preferred, with ascorbyl palmitate being especially preferred.

[0015] Suitable industrial emulsifiers are, for example, lauryl ethylene oxide (LEO)-9 and (LEO)-10.

[0016] The method in accordance with the invention is especially suitable for the production of liquid dispersions from oils, such as, for example, corn oil, palm oil, sunflower oil and the like; and liquid dispersions from lipophilic active substances, such as, for example, from vitamin A, D, E and K, from carotenoids or from food additives such as PUFAs and tocotrienols.

[0017] Suitable carotenoids are, for example, beta-carotene, beta-apo-4'-carotenal, beta-apo-8'-carotenal, beta-apo-12'-carotenal, beta-apo-8'-carotenoic acid, astaxanthin, canthaxanthin, zeaxanthin, cryptoxanthin, citranaxanthin, lutein, lycopene, torularodin aldehyde, torularodin ethyl ester, neurosporaxanthin ethyl ester, zetacarotene, dehydro-plectania-xanthin and the like.

[0018] The aforementioned lipophilic active substances can be used directly insofar as they are oily substances. Solid active substances, for example carotenoids, are used in dissolved form in oil or in water-immiscible solvents.

[0019] Suitable water-immiscible solvents are halogenated aliphatic hydrocarbons such as e.g. methylene chloride, water-immiscible esters such as carboxylic acid dimethyl ester (dimethyl carbonate), ethyl formate, methyl, ethyl or iso-propyl acetate; or water-immiscible ethers such as e.g. methyl tert.butyl ether and the like.

[0020] The process in accordance with the invention provides a very efficient mixing or dispersing process for liquids.

[0021] The mixing or dispersing process in accordance with the invention is also suitable for performing chemical reactions having very short reaction times, in the region of seconds or fractions of seconds.

[0022] The mixing device in accordance with the invention has, in contrast to the known devices described hereinbefore, an arrangement of the bores of the inlet and outlet nozzles which is axial to one another. Thereby and by the turbulence chamber positioned between the nozzles, the short term stability of mixtures, especially of dispersions, is increased. This results in the liquid dispersion being homogenized more strongly.

[0023] The invention will be illustrated hereinafter on the basis of the Figures.

Fig. 1 shows a flow scheme of an arrangement for performing the method in accordance with the invention,

Fig. 2 shows a cross section through a mixing device in accordance with the invention having an inlet nozzle and an outlet nozzle,

Fig. 3 shows a perspective view of the mixing device in accordance with the invention,

Fig. 4 shows a possibility of a *scale-up* arrangement,

Fig. 5 shows a further possibility of a *scale-up* arrangement.

[0024] In Fig. 1 a supply container (1) is followed by a high pressure pump (2) which is optionally connected to a heat exchanger (3). The mixing device (4) is positioned thereafter.

[0025] Fig. 2 and Fig. 3 show a mixing device (4) consisting of an inlet nozzle (6) having a bore diameter of about 0.05 mm to about 1 mm, preferably about 0.05 mm to about 0.5 mm; a turbulence chamber (7) having a diameter of about 0.5 mm to about 10 mm, preferably about 1 mm to about 10 mm, especially about 1 mm to about 5 mm; an outlet nozzle (8) having a bore diameter of about 0.05 mm to about 1.5 mm, preferably about 0.05 mm to about 0.8 mm, with the inlet nozzle (6), the turbulence chamber (7) and the outlet nozzle (8) being pressed in sequence in a cylindrical support (5). The axes of the bores of the inlet nozzle and the outlet nozzle are arranged axially to one another.

[0026] The ratio of length to diameter of the nozzle bore amounts in the case of the inlet nozzle or the outlet nozzle to about 1 to 10, preferably about 1 to 5.

[0027] The ratio of length to diameter of the turbulence chamber is about 0.5 to about 50, preferably about 0.5 to about 20, especially about 0.5 to about 10.

[0028] The diameter of the turbulence chamber must be greater than the diameter of the outlet nozzle.

[0029] The bore diameters of the inlet nozzle and the outlet nozzle can be the same or different. However, an embodiment in which the bore diameter of the inlet nozzle is smaller than the bore diameter of the outlet nozzle is preferred. For example, the bore diameter of the inlet nozzle is about 0.2 mm and the bore diameter of the outlet nozzle is about 0.25 mm.

[0030] The nozzles are suitably manufactured from wear-resistant materials such as e.g. sapphire, diamond, stainless steel, ceramic, silicon carbide, tungsten carbide, zirconium or the like.

[0031] The bores of the nozzles can be round or rectangular or can have the form of an ellipse. A bore which has a cone in the mouth is also suitable.

[0032] The support (5) likewise consists of wear-resistant materials, suitably of stainless steel.

[0033] Fig. 4 shows one possibility for the scale-up of the mixing device (4).

[0034] Section 4a shows a plurality of nozzles in accordance with the invention with insert (11), which are screwed into a support plate (10). The support plate is positioned in a conduit (9).

[0035] Cross section 4b shows only one nozzle insert (11'). The nozzle insert (11'), the support plate (10) as well as the conduit (9) are manufactured from wear-resistant materials, preferably stainless steel.

[0036] Section 4c shows the screwable nozzle support (11") which contains the nozzle in accordance with the invention.

[0037] Fig. 5 shows a further *scale-up* possibility. The mixing device consisting of a support disk (12), a turbulence chamber (13) and a support disk (14), which are positioned in sequence in a tubular conduit (15), with the first support disk (12) containing a plurality of inlet nozzles (16) having a bore diameter of about 0.05 mm to about 1 mm, preferably about 0.05 mm to about 0.5 mm, and the second support disk (14) containing a plurality of outlet nozzles (17) having a bore diameter of about 0.05 mm to about 1 mm, preferably about 0.05 mm to about 0.8 mm. The axes of the nozzles (16) and (17) are arranged axially to one another.

[0038] The number of nozzles determines the diameter of the turbulence chamber (13). The ratio of length to diameter of the turbulence chamber is designed such that the residence time of the liquid to be dispersed in the dispersing unit is about 10^{-6} sec to about 10^{-1} sec.

[0039] For the production of a finely divided liquid dispersion, set forth in Fig. 1, a pre-emulsion is firstly produced in the supply container (1) in a known manner and pumped through the dispersing unit (4) at temperatures of about 20°C to about 250°C, preferably about 20°C to about 200°C, and pressures of about 50 bar to about 2500 bar, preferably about 50 bar to about 800 bar, using a high pressure pump (2). Where required, the pre-emulsion can be heated for a brief period in the heat exchanger (3). The residence time of the liquid to be dispersed in the dispersing unit is about 10^{-6} sec to about 10^{-1} sec.

[0040] The following Examples illustrate the invention in more detail, but are not intended to limit its scope. In the Examples there was also used, in addition to the food emulsifier ascorbyl palmitate, the industrial emulsifiers lauryl ethylene oxide (LEO)-9 and (LEO)-10. This is a so-called "more rapid" emulsifier which very rapidly stabilizes newly formed phase boundaries.

Example 1: Corn oil and lauryl ethylene oxide

[0041] The emulsion had the following composition:

87 wt.% deionized water, 10 wt.% corn oil and 3 wt.% lauryl ethylene oxide-9.

[0042] Deionized water was placed in a kettle and warmed to 40°C. The emulsifier lauryl ethylene oxide (LEO)-9 was dissolved in the water. Subsequently, the corn oil was stirred in and comminuted with an Ultra Turrax mixer at 1000 rpm. When the content of dispersed phase was 10 wt.%, the weight ratio of corn oil to lauryl ethylene oxide was 10:3. The pre-emulsion was homogenized three times at a pressure of 600 bar using the dispersing unit according to Fig. 2 in accordance with the invention. The geometric dimensions of the dispersing units used are given in Table 1. The particle sizes were determined in a known manner by means of photon correlation spectroscopy.

Example 2: Corn oil and ascorbyl palmitate.

[0043] Here, ascorbyl palmitate was used as the emulsifier. The quantitative composition of the emulsion corresponded to that in Example 1.

[0044] Deionized water was placed in a kettle and warmed to 40°C. Ascorbyl palmitate was dissolved in the water at pH values between seven and eight. The production of the pre-emulsion and the homogenization were carried out according to Example 1.

Example 3: dl-alpha-Tocopherol and ascorbyl palmitate.

[0045] Example 3 was carried out in accordance with Example 2.

Example 4: dl-alpha-Tocopherol and ascorbyl palmitate

[0046] A pre-emulsion was produced in accordance with Example 2. The content of dispersed phase was 30 wt.%. The weight ratio of dl-alpha-tocopherol to ascorbyl palmitate was 10:1. The pre-emulsion was homogenized once at pressures of 100 bar, 200 bar, 300 bar, 400 bar and 500 bar using the dispersing unit in accordance with the invention shown in Fig. 2.

Example 5: dl-alpha-Tocopherol, corn oil with ascorbyl palmitate and fish gelatine

[0047] An emulsion comprising 65 wt.% deionized water, 6 wt.% ascorbyl palmitate, 4 wt.% fish gelatine, 18 wt.% dl-alpha-tocopherol and 7 wt.% corn oil was produced in the manner described hereinafter.

[0048] The deionized water was placed in a kettle and warmed to 60°C. The fish gelatine was dissolved in the water. Then, the ascorbyl palmitate was dissolved in the aforementioned solution at pH values between seven and eight. Subsequently, the dispersed phase consisting of dl-alpha-tocopherol and corn oil was stirred in as described in Example 1. The pre-emulsion was homogenized in accordance with Example 4.

[0049] Examples 6-10 are comparative Examples using a single-hole nozzle.

Example 6: Corn oil and lauryl ethylene oxide

[0050] The pre-emulsion was produced in accordance with Example 1 and homogenized three times at a pressure of 600 bar in a single-hole nozzle, said single-hole nozzle referring to a nozzle having an acute angled inlet and outlet. The geometric dimensions of the single-hole nozzle are given in Table 1.

Example 7: Corn oil and ascorbyl palmitate

[0051] The pre-emulsion was produced in accordance with Example 2 and homogenized in the manner described in Example 6.

Example 8: dl-alpha-Tocopherol and ascorbyl palmitate

[0052] The pre-emulsion was produced in accordance with Example 3 and homogenized in the manner described in Example 6.

Example 9: dl-apha-Tocopherol and ascorbyl palmitate

[0053] The pre-emulsion was produced in accordance with Example 4 and homogenized once in a single-hole nozzle as described in Example 6 at pressures of 100 bar, 200 bar, 300 bar, 400 bar and 500 bar. The particle size was determined in a known manner by means of laser diffraction spectrometry and photon correlation spectroscopy.

Example 10: dl-alpha-Tocopherol, corn oil with ascorbyl palmitate and fish gelatine

[0054] The pre-emulsion was produced in accordance with Example 5 and homogenized once in a single-hole nozzle as described in Example 6 at pressures of 100 bar, 200 bar, 300 bar, 400 bar and 500 bar.

Table 1

Geometric dimensions of the dispersing units used.				
Nozzle type	Bore diameter of the inlet nozzle [mm]	Bore diameter of the turbulence chamber [mm]	Length of the turbulence chamber [mm]	Bore diameter of the outlet nozzle [mm]
Nozzle I	0.2	2	1.5	0.25
Nozzle I long	0.2	2	3	0.28
Nozzle II	0.2	2	1.5	0.2
single-hole nozzle	0.2	-	-	-

[0055] The average particle sizes of the finely divided liquid dispersions of Examples 1-6 obtained are set forth in Tables 2 and 3.

Table 2

Average particle sizes in nm of experiments 1, 2, 3, 6, 7 and 8.				
Example	Nozzle type	Passage 1 Particle size	Passage 2 Particle size	Passage 3 Particle size
1	Nozzle I 0.2/0.25 mm	218 219	202 215	202 200
1	Nozzle II 0.2/0.2 mm	230 231	214 220	212 208
6	single-hole nozzle 0.2 mm	307 298	256 250	247 248
2	Nozzle I 0.2/0.25 mm	275	250	238
2	Nozzle II 0.2/0.2 mm	294	266	245
7	single-hole nozzle 0.2 mm	340	320	275
3	Nozzle I 0.2/0.25 mm	295	287	267
3	Nozzle II 0.2/0.2 mm	312	294	302
8	single-hole nozzle 0.2 mm	442	416	403

[0056] From Table I it will be evident that the homogenization using nozzles I and II in accordance with the invention gives a liquid dispersion with a smaller particle size compared with the homogenization using a single-hole nozzle. When nozzles I and II are used, particle sizes up to a third smaller are produced compared with the single-hole nozzle.

[0057] The best homogenization takes place in nozzle I. Here, the particle size was reduced to 200 nm after a triple homogenization. The values from Example 1 reveal that the reproducibility of the results is also good.

[0058] The average particle sizes of the finely divided liquid dispersions obtained in Examples 4,5,9 and 10 are set forth in Table 3.

Table 3

Average particle sizes in nm of experiments 4, 5, 9 and 10.						
Ex.	Nozzle type	100 bar Particle size [nm]	200 bar Particle size [nm]	300 bar Particle size [nm]	400 bar Particle size [nm]	500 bar Particle size [nm]
4	Nozzle I 0.2/0.25 mm	1800	1370	1400	1105	1080
4	Nozzle I/long 0.2/0.28 mm	1370	740	745	660	600
9	single-hole noz-zle 0.2 mm	5200	3080	1520	1370	914
5	Nozzle I 0.2/0.25 mm	435	410	340	350	345
5	Nozzle I/long 0.2/0.28 mm	420	410	360	325	290

Table 3 (continued)

Average particle sizes in nm of experiments 4, 5, 9 and 10.						
Ex.	Nozzle type	100 bar Particle size [nm]	200 bar Particle size [nm]	300 bar Particle size [nm]	400 bar Particle size [nm]	500 bar Particle size [nm]
10	single-hole nozzle 0.2 mm	440	430	360	350	355

[0059] From Table 3 it will be evident that the homogenization using nozzle I and nozzle I/long give liquid dispersions with a smaller particle size than the homogenization using a single-hole nozzle. The best homogenization takes place using nozzle I/long.

Claims

1. A method for mixing or dispersing liquids, which method comprises pumping the liquids to be mixed or to be dispersed at temperatures of about 20°C to about 250°C, preferably of about 20°C to about 200°C, and pressures of about 50 bar to about 2500 bar, preferably of about 100 bar to about 800 bar, through a mixing device which consists of one or more inlet nozzles, one or more turbulence chambers and one or more outlet nozzles, with the inlet nozzle(s), turbulence chamber(s) and outlet nozzle(s) being pressed in sequence in a cylindrical support and the axes of the bores of the inlet nozzle and the outlet nozzle being arranged axially to one another.

2. A method in accordance with claim 1, wherein liquids are dispersed.

3. A method in accordance with claim 1 or claim 2, wherein the residence time of the liquid to be mixed or to be dispersed in the mixing device is about 10^{-6} sec to about 10^{-1} sec.

4. A method in accordance with any one of claims 1 to 3, wherein a liquid dispersion having an average particle size of about 10 nm to about 1000 nm, preferably about 50 nm to about 400 nm, is produced.

5. A method in accordance with any one of claims 1 to 4, wherein a pre-emulsion is pumped at about 20°C to about 200°C and pressures of about 50 bar to about 2500 bar, preferably of about 100 bar to about 800 bar, through a mixing device (4).

6. A method in accordance with claim 5, wherein the pre-emulsion is an oil-in-water emulsion in which the viscosity of the dispersed phase is about 0.01 mPas to about 10,000 mPas, preferably about 0.1 mPas to about 2000 mPas.

7. A method in accordance with claim 5 or claim 6, wherein the pre-emulsion is produced by stirring the liquid to be dispersed into an aqueous emulsifier solution, optionally while warming.

8. A method in accordance with claim 7, wherein the emulsifier is ascorbyl palmitate.

9. A method in accordance with any one of claims 1 to 8, wherein oils or lipophilic active substances are dispersed.

10. A method in accordance with claim 9, wherein the lipophilic active substances are vitamins A, D, E and K, carotenoids or food additives such as PUFAs and tocotrienols.

11. A mixing device for performing the method according to any one of claims 1 to 10, said device consisting of an inlet nozzle (6), a turbulence chamber (7) and an outlet nozzle (8), with the inlet nozzle (6), the turbulence chamber (7) and the outlet nozzle (8) being pressed in sequence in a cylindrical support (5) and the axes of the bores of the inlet nozzle and the outlet nozzle being arranged axially to one another.

12. A device according to claim 11, wherein the bore diameter of the inlet nozzle (6) is about 0.05 mm to about 1 mm, preferably about 0.05 mm to about 0.5 mm, the diameter of the turbulence chamber (7) is about 0.5 mm to about 10 mm, preferably about 1 mm to about 5 mm, especially about 1 mm to about 5 mm, and the bore diameter of the outlet nozzle (8) is about 0.05 mm to about 1.5 mm, preferably about 0.05 mm to about 0.8 mm.

13. A device according to claim 11 or claim 12, wherein the bore diameter of the inlet nozzle is smaller than the bore

diameter of the outlet nozzle.

14. A device according to any one of claims 11 to 13, wherein the nozzles are manufactured from sapphire, diamond, stainless steel, ceramic, silicon carbide, tungsten carbide or zirconium oxide.

15. A device according to any one of claims 11 to 14, wherein the ratio of length to diameter of the nozzle bores in the inlet nozzle and the outlet nozzle is about 1 to about 10, preferably about 1 to about 5.

16. A device according to any one of claims 11 to 15, wherein the ratio of length to diameter of the turbulence chamber is about 0.5 to about 50, preferably about 0.5 to about 20, especially about 0.5 to about 10.

17. A device according to any one of claims 11 to 16, wherein the diameter of the turbulence chamber is greater than the diameter of the outlet nozzle.

18. A mixing device for performing the method according to any one of claims 1 to 10, in which a plurality of nozzles in accordance with the invention with an insert (11) are screwed into a support plate (10) and the support plate (10) is positioned in a conduit.

19. A mixing device for performing the method according to any one of claims 1 to 10, said device consisting of a support disk (12), a turbulence chamber (13) and a support disk (14), which are positioned in sequence in a conduit (15), the first support disk (12) containing a plurality of inlet nozzles (16) having a bore diameter of about 0.05 mm to about 1 mm, preferably about 0.05 mm to about 0.5 mm, and the second support disk (14) containing a plurality of outlet nozzles having a bore diameter of about 0.05 mm to about 1 mm, preferably about 0.05 mm to about 0.8 mm, and the axes of the nozzles (16) and (17) being arranged axially to one another.

20. The use of a mixing device in accordance with any one of claims 11-19 for mixing or dispersing liquids.

21. The use of a mixing device in accordance with any one of claims 11-19 for the performance of chemical reactions with very short reaction times in the region of seconds or fractions of seconds.

Fig. 1:

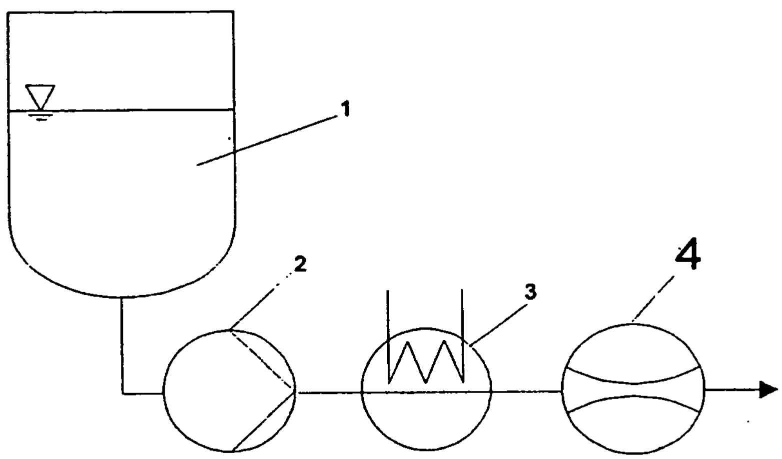


Fig. 2:

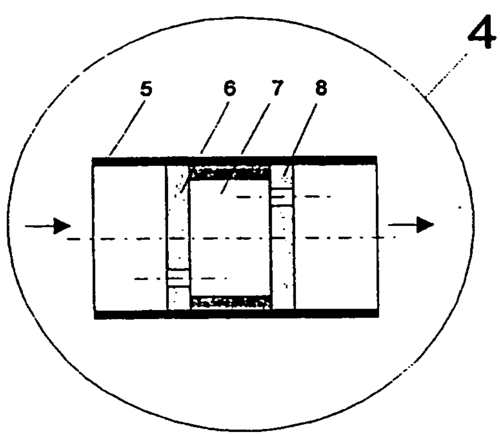


Fig. 3:

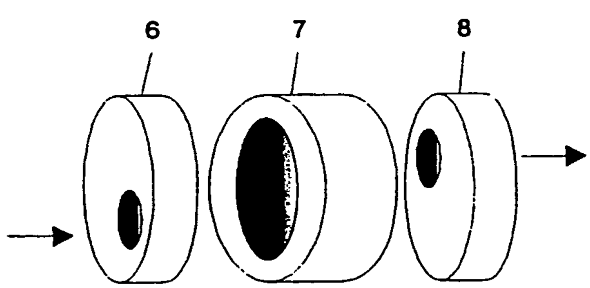
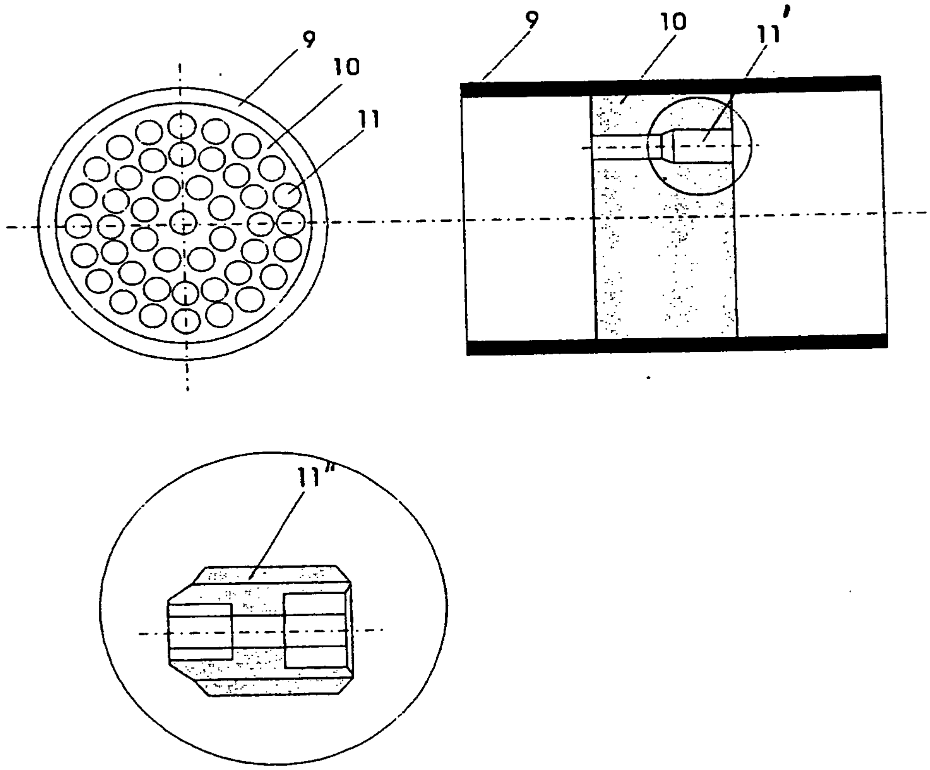


Fig. 4:

4a

4b



4c

Fig. 5: